Docket No. 1422-0603P
(PATENT)

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In Re Application of:	:	Confirmation No.: 1568
Yasuhiro YONEDA et al.	:	Group Art Unit: 1755
Serial No. 10/668,216	:	Examiner: MARCHESCHI,
Filed: September 24, 2003	:	Michael A.
For: POLISHING COMPOSITION		

DECLARATION UNDER 37 C.F.R. 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Yasuhiro YONEDA, residing at Wakayama-ken, Japan, hereby declare and state as follows:

1. That I am one of the co-inventors of U.S. Application Serial No. 10/668,216 filed on September 24, 2003. I am thoroughly familiar with the contents of said Application, its prosecution before the United States Patent and Trademark Office and the references cited therein.

2. That I am a graduate of Osaka University, Department of Engineering and received a master's degree in the year 1990, majoring in applied chemistry.

3. That I have been employed in Kao Corporation in the year 1990 and have been assigned to the Research Laboratories.

4. That I have been involved in the research and development of polishing composition since the year 1999.

5. That the following experiments were conducted by myself or under my direct supervision and control in order to verify that the polishing composition containing a colloidal silica and polymer particles as described in the present invention has excellent polishing rate and excellent dispersibility as compared to conventional polishing composition without containing a colloidal silica and polymer particles.

EXPERIMENTAL METHOD

I. Polishing Test

A polishing composition was prepared using the silica slurry shown in Table I (Kind 8 or 9), and the polymer particles (e) described in the present specification (average particle size: 138 nm). A polishing test was conducted with the polishing composition prepared. The experimental procedures are the same as those described in Example 1 of the present specification as detailed below.

Preparation of Polishing Composition

The ion-exchanged water was added to and mixed with an aqueous dispersion of the polymer particles (e) obtained in the manner detailed below, out of which the polymer particles constitute 3 parts, with stirring. In an agitated state, an aqueous dispersion of silica was added to and mixed with the resulting mixture, out of which the inorganic particles constitute 13 parts, to give a polishing composition. As occasion demands, the pH of the polishing composition was adjusted with an aqueous potassium hydroxide to 10.5 to 11.5.

Preparation of Polymer Particles (e)

There were mixed together 30 parts of styrene, 1.5 parts of a potassium salt of a fatty acid (commercially available from Kao Corporation under the trade name of KS SOAP), 0.06 parts of potassium persulfate and 60 parts of ion-exchanged water, with a homomixer with stirring, to give a monomer emulsion. Next, a 2-L separable flask was charged with 8.5 parts of ion-exchanged water, 0.017 parts of potassium persulfate, and 4.6 parts of the previously prepared monomer emulsion, and the air inside the flask was substituted with nitrogen gas, and the temperature was raised to 80°C to react the monomer emulsion. Thereafter, the remaining 86.9 parts of the previously prepared monomer emulsion was fed to the flask at a given rate over a period of 5 hours, to give an aqueous dispersion of the polymer particles. The polymer particles had an average particle size of 138 nm.

The polishing test was carried out using the polishing composition as prepared above under the following conditions, and evaluated.

(1) Polishing Conditions

As a substrate to be polished, there was used one in which silicon oxide film is formed on an 8-inch (200 mm) silicon substrate in a thickness of 2000 nm by plasma TEOS method, and the resulting substrate was cut into a piece having 40 mm each side. As the polishing machine, there was used a single-sided polishing machine (product number: MA-300, commercially available from Musasino Denshi K.K.). As the polishing pad, there was used IC-1000 050(P) Type 52/S400 12"PJ (trade name, commercially available from RODEL NITTA K.K.). In addition, the polishing load was 39.2 kPa, and the feed amount of the polishing composition was 50 ml/min. The number of rotations of the disc was

90 r/min, and the number of rotations of the head was 90 r/min, and the disc and the head were rotated unidirectionally. The polishing time was 2 minutes.

(2) Calculation of Polishing Rate

The polishing rate was obtained by carrying out polishing under the above-mentioned conditions, determining a thickness of a silicon oxide film on the substrate to be polished before and after polishing, and dividing the thickness by the polishing time as expressed by the following equation:

Polishing Rate (nm/min) =[Thickness (nm) Before Polishing – Thickness (nm) After Polishing]/Polishing Time (min)

The film thickness was determined with a spectrometric film thickness measuring system (trade name: LAMBDA ACE VM-1000, commercially available from DAINIPPON SCREEN MFG. CO., LTD.).

II. Dispersibility Test

The same procedures as in the above Experimental Method I. were carried out with the silica slurry shown in Table I (Kind 5, 7, 8 or 9), and the polymer particles (e) described above, to give polishing compositions having different pHs, the composition containing silica (13% by weight) and polymer particles (3% by weight). In the pH adjustment, an aqueous potassium hydroxide or nitric acid was used as occasion demands. Eighty grams of the resulting polishing composition was placed in a 110 ml screw pipe (manufactured by K.K. Maruemu), and the screw pipe was allowed to stand at 20°C for 10 days. The aggregation state of the polishing composition after having allowed to stand was visually observed.

Table I

	Kind	Trade Name	Manufacturer	Average Particle Size (nm)	Solid Ingredient (% by wt.)
(1)	Colloidal Silica	Cataloid SI-30	CATALYSTS & CHEMICALS INDUSTRIES CO., LTD.	11	30
(2)	Colloidal Silica	Cataloid SI-50	CATALYSTS & CHEMICALS INDUSTRIES CO., LTD.	26	50
(3)	Colloidal Silica	Cataloid SI-45P	CATALYSTS & CHEMICALS INDUSTRIES CO., LTD.	45	40
(4)	Colloidal Silica	Levasil 50CK	Bayer Ltd.	85	30
(5)	Colloidal Silica	Spherica Slurry 160	CATALYSTS & CHEMICALS INDUSTRIES CO., LTD.	160	16
(6)	Colloidal Silica	Syton HF-50F	Du Pont Kabushiki Kaisha	45	40
(7)	Colloidal Silica	Spherica Slurry 120	CATALYSTS & CHEMICALS INDUSTRIES CO., LTD.	120	18
(8)	Colloidal Silica	MP-2040	Nissan Chemical Industries, Ltd.	200	40
(9)	Fumed Silica	Semi-Sperse 25	Cabot Microelectronics	170 [1)]	25

Note 1) Value determined by light scattering method using an electrophoretic light scattering (ELS) spectrophotometer (commercially available from Otsuka Electronics Co., Ltd. under the trade name of Electrophoretic Light Scattering Spectrophotometer (Laser Zeta Potentiometer) ELS8000)

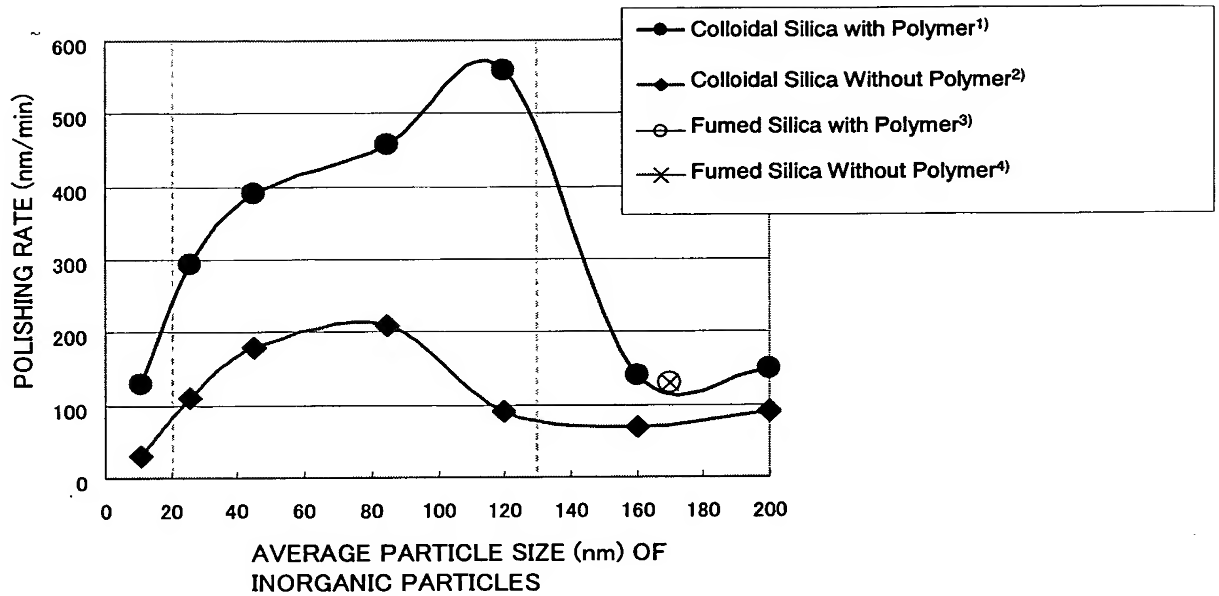
RESULTS

The results of the above Polishing Test are shown in Table II. Also, the results of the Polishing Test and those test results described in the specification are summarized in a graph shown in Figure A. In addition, the results of the Dispersibility Test are shown in Table III.

Table II

Polishing Test	Inorganic Particles				Polymer Particles			Di + 50 nm	Ratio of Contents (Cp/Ci)	Polishing Rate (nm/min)
		Kind	Average Particle Size Di (nm)	Content (Effective Ingredient) (% by wt.)	Kind	Average Particle Size Dp (nm)	Content (Effective Ingredient) (% by wt.)			
Follow-up Test 1	(8)	Colloidal	200	13	e	138	3	250	0.23	150
Follow-up Test 2	(8)	Colloidal	200	13	–	–	–	–	–	90
Follow-up Test 3	(9)	Fumed	170	13	e	138	3	220	0.23	130
Follow-up Test 4	(9)	Fumed	170	13	–	–	–	–	–	130

FIG. A



Note: 1) Average value of polishing rate for each particle size in Examples 1-20 and Follow-up Test 1.
2) Polishing rate for each particle size in Comparative Examples 1-6 and Follow-up Test 2.
3) Polishing rate for each particle size in Follow-up Test 3.
4) Polishing rate for each particle size in Follow-up Test 4.

Table III

pH	Colloidal Silica			Fumed Silica
	Kind (7) Average Particle Size: 120 nm	Kind (5) Average Particle Size: 160 nm	Kind (8) Average Particle Size: 200 nm	Kind (9) Average Particle Size: 170 nm
2	Not gelated	Not gelated	Not gelated	Gelated
7-8	Not gelated	Not gelated	Not gelated	Not gelated
11	Not gelated	Not gelated	Not gelated	Not gelated

DISCUSSION

I. Polishing Test

As shown in Comparative Examples 5 and 6 of the present specification and Follow-up test 2, when the polishing composition contains only the colloidal silica as an abrasive, the polishing rate is more likely to be lowered as the particle sizes become larger. Also, in Follow-up Test 1, the same polymer particles as in Example 6 of the present specification are contained in the same amount, but an effect of increasing the polishing rate by a combined use of the colloidal silica and the polymer particles is small as in Example 7 of the present specification (presently comparative example). In other words, it can be seen that even if a polishing composition contains colloidal silicas having particles sizes larger than the claimed size range, a high polishing rate cannot be obtained. Also, it can be seen from Comparative Example 1 and Example 1 (presently comparative example) of the present specification that a polishing composition contains colloidal silica smaller than the claimed size range, a high polishing rate cannot be obtained.

In addition, when the fumed silica is used, an effect of increasing the polishing rate cannot be confirmed. Therefore, the effect of the present invention is remarkable in the colloidal silica.

It was clarified from the above that when the colloidal silica having an average size of from 20 to 130 nm and the polymer particles are used in combination, a surprisingly remarkably excellent effect that the polishing rate of the resulting polishing composition is remarkably increased as compared to the case where the colloidal silica and the polymer particles are not used in combination is exhibited.

II. Dispersibility Test

As shown in Table III, the polishing composition containing fumed silica as an abrasive is gelated at a pH of 2. On the other hand, the polishing composition containing colloidal silica as an abrasive shows excellent dispersibility without being gelated in any of particle size ranges including a particle size of 200 nm and any pHs.. It can be seen from the above that the colloidal silica is excellent in dispersibility as compared to the fumed silica even when the colloidal silicas have larger particle sizes than the fumed silica.

It is clarified from the above that when the polishing composition contains a colloidal silica as an abrasive, an excellent effect that a stable polishing composition is obtained over a long period of time is exhibited.

6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

7. Further declarant saith not.

Yasuhiro Yoneda

Yasuhiro YONEDA

Feb. 3, 2006

Date